

February 18, 2016

Tom Moe
USS Corporation
P.O. Box 417
Mountain Iron, MN 55768

RE: Project: NPDES-LINE 3 Wk1
Pace Project No.: 1260694

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on February 04, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,



Heather R Zika
heather.zika@pacelabs.com
Project Manager

Enclosures

cc: Terri Sabetti, NTS



REPORT OF LABORATORY ANALYSIS

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CERTIFICATIONS

Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792

Alaska Certification #MN01084

Arizona Department of Health Certification #AZ0785

Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification # : 998027470

WA Department of Ecology Lab ID# C1007

Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality

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SAMPLE SUMMARY

Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

| Lab ID | Sample ID | Matrix | Date Collected | Date Received |
|------------|--------------------------|--------|----------------|----------------|
| 1260694001 | WS-003 Thckener Overflow | Water | 02/03/16 08:40 | 02/04/16 16:48 |
| 1260694002 | WS-002 Scrubber Make-Up | Water | 02/03/16 08:50 | 02/04/16 16:48 |
| 1260694003 | WS-003 Thckener Overflow | Water | 02/03/16 08:40 | 02/04/16 16:48 |

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SAMPLE ANALYTE COUNT

Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

| Lab ID | Sample ID | Method | Analysts | Analytes Reported | Laboratory |
|------------|--------------------------|-----------|----------|-------------------|------------|
| 1260694001 | WS-003 Thckener Overflow | EPA 300.0 | DMB | 2 | PASI-V |
| 1260694002 | WS-002 Scrubber Make-Up | EPA 200.7 | MAR | 3 | PASI-V |
| | | EPA 300.0 | DMB | 1 | PASI-V |
| 1260694003 | WS-003 Thckener Overflow | EPA 200.7 | MAR | 3 | PASI-V |
| | | EPA 300.0 | DMB | 1 | PASI-V |

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ANALYTICAL RESULTS

Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

| Sample: WS-003 Thckener Overflow Lab ID: 1260694001 Collected: 02/03/16 08:40 Received: 02/04/16 16:48 Matrix: Water | | | | | | | | | |
|--|------------|-------|--------------|------|----|----------|----------------|------------|------|
| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
| 300.0 IC Anions 28 Days Analytical Method: EPA 300.0 | | | | | | | | | |
| Chloride | 256 | mg/L | 10.0 | 5.0 | 10 | | 02/09/16 03:30 | 16887-00-6 | |
| Fluoride | 3.1 | mg/L | 1.0 | 0.24 | 10 | | 02/09/16 03:30 | 16984-48-8 | |

| Sample: WS-002 Scrubber Make-Up Lab ID: 1260694002 Collected: 02/03/16 08:50 Received: 02/04/16 16:48 Matrix: Water | | | | | | | | | |
|---|-------------|-------|--------------|------|----|----------------|----------------|------------|------|
| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
| 200.7 MET ICP, Lab Filtered Analytical Method: EPA 200.7 Preparation Method: EPA 200.7 | | | | | | | | | |
| Calcium, Dissolved | 46.6 | mg/L | 5.0 | 0.29 | 10 | 02/10/16 16:53 | 02/11/16 10:47 | 7440-70-2 | |
| Magnesium, Dissolved | 217 | mg/L | 5.0 | 0.67 | 10 | 02/10/16 16:53 | 02/11/16 10:47 | 7439-95-4 | |
| Total Hardness, Dissolved | 1010 | mg/L | 100 | 50.0 | 10 | 02/10/16 16:53 | 02/11/16 10:47 | | |
| 300.0 IC Anions 28 Days Analytical Method: EPA 300.0 | | | | | | | | | |
| Sulfate | 850 | mg/L | 20.0 | 0.89 | 10 | | 02/09/16 03:52 | 14808-79-8 | |

| Sample: WS-003 Thckener Overflow Lab ID: 1260694003 Collected: 02/03/16 08:40 Received: 02/04/16 16:48 Matrix: Water | | | | | | | | | |
|--|-------------|-------|--------------|------|----|----------------|----------------|------------|------|
| Parameters | Results | Units | Report Limit | MDL | DF | Prepared | Analyzed | CAS No. | Qual |
| 200.7 MET ICP, Lab Filtered Analytical Method: EPA 200.7 Preparation Method: EPA 200.7 | | | | | | | | | |
| Calcium, Dissolved | 395 | mg/L | 5.0 | 0.29 | 10 | 02/10/16 16:53 | 02/11/16 10:50 | 7440-70-2 | |
| Magnesium, Dissolved | 109 | mg/L | 5.0 | 0.67 | 10 | 02/10/16 16:53 | 02/11/16 10:50 | 7439-95-4 | |
| Total Hardness, Dissolved | 1430 | mg/L | 100 | 50.0 | 10 | 02/10/16 16:53 | 02/11/16 10:50 | | |
| 300.0 IC Anions 28 Days Analytical Method: EPA 300.0 | | | | | | | | | |
| Sulfate | 1300 | mg/L | 40.0 | 1.8 | 20 | | 02/09/16 04:14 | 14808-79-8 | |

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QUALITY CONTROL DATA

Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

QC Batch: MPRP/6472

Analysis Method: EPA 200.7

QC Batch Method: EPA 200.7

Analysis Description: 200.7 MET Dissolved

Associated Lab Samples: 1260694002, 1260694003

METHOD BLANK: 287268

Matrix: Water

Associated Lab Samples: 1260694002, 1260694003

| Parameter | Units | Blank Result | Reporting Limit | MDL | Analyzed | Qualifiers |
|----------------------|-------|--------------|-----------------|-------|----------------|------------|
| Calcium, Dissolved | mg/L | ND | 0.50 | 0.029 | 02/11/16 10:08 | |
| Magnesium, Dissolved | mg/L | ND | 0.50 | 0.067 | 02/11/16 10:08 | |

LABORATORY CONTROL SAMPLE: 287269

| Parameter | Units | Spike Conc. | LCS Result | LCS % Rec | % Rec Limits | Qualifiers |
|----------------------|-------|-------------|------------|-----------|--------------|------------|
| Calcium, Dissolved | mg/L | 50 | 51.9 | 104 | 85-115 | |
| Magnesium, Dissolved | mg/L | 50 | 51.6 | 103 | 85-115 | |

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 287270

287271

| Parameter | Units | 1260723001 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|----------------------|-------|-------------------|----------------|-----------------|-----------|------------|----------|-----------|--------------|-----|---------|------|
| Calcium, Dissolved | mg/L | 318 | 50 | 50 | 369 | 373 | 101 | 110 | 70-130 | 1 | 20 | |
| Magnesium, Dissolved | mg/L | 117 | 50 | 50 | 166 | 170 | 100 | 107 | 70-130 | 2 | 20 | |

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 287272

287273

| Parameter | Units | 1260761001 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|----------------------|-------|-------------------|----------------|-----------------|-----------|------------|----------|-----------|--------------|-----|---------|------|
| Calcium, Dissolved | mg/L | 60.4 | 50 | 50 | 111 | 111 | 100 | 101 | 70-130 | 0 | 20 | |
| Magnesium, Dissolved | mg/L | 61.4 | 50 | 50 | 111 | 111 | 100 | 100 | 70-130 | 0 | 20 | |

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

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QUALITY CONTROL DATA

Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

QC Batch: WETA/15597 Analysis Method: EPA 300.0
QC Batch Method: EPA 300.0 Analysis Description: 300.0 IC Anions
Associated Lab Samples: 1260694001, 1260694002, 1260694003

METHOD BLANK: 286523 Matrix: Water

Associated Lab Samples: 1260694001, 1260694002, 1260694003

| Parameter | Units | Blank Result | Reporting Limit | MDL | Analyzed | Qualifiers |
|-----------|-------|--------------|-----------------|-------|----------------|------------|
| Chloride | mg/L | ND | 1.0 | 0.50 | 02/08/16 20:32 | |
| Fluoride | mg/L | ND | 0.10 | 0.024 | 02/08/16 20:32 | |
| Sulfate | mg/L | ND | 2.0 | 0.089 | 02/08/16 20:32 | |

LABORATORY CONTROL SAMPLE: 286524

| Parameter | Units | Spike Conc. | LCS Result | LCS % Rec | % Rec Limits | Qualifiers |
|-----------|-------|-------------|------------|-----------|--------------|------------|
| Chloride | mg/L | 50 | 50.5 | 101 | 90-110 | |
| Fluoride | mg/L | 5 | 5.1 | 101 | 90-110 | |
| Sulfate | mg/L | 50 | 49.3 | 99 | 90-110 | |

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 286525 286526

| Parameter | Units | 1260684001 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|-----------|-------|-------------------|----------------|-----------------|-----------|------------|----------|-----------|--------------|-----|---------|------|
| Chloride | mg/L | 114 | 50 | 50 | 164 | 164 | 98 | 98 | 90-110 | 0 | 20 | |
| Fluoride | mg/L | ND | 5 | 5 | 4.8 | 4.8 | 95 | 95 | 90-110 | 0 | 20 | |
| Sulfate | mg/L | 103 | 50 | 50 | 153 | 153 | 100 | 100 | 90-110 | 0 | 20 | |

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 286527 286528

| Parameter | Units | 1260737004 Result | MS Spike Conc. | MSD Spike Conc. | MS Result | MSD Result | MS % Rec | MSD % Rec | % Rec Limits | RPD | Max RPD | Qual |
|-----------|-------|-------------------|----------------|-----------------|-----------|------------|----------|-----------|--------------|-----|---------|------|
| Chloride | mg/L | 68.2 | 500 | 500 | 582 | 583 | 103 | 103 | 90-110 | 0 | 20 | |
| Fluoride | mg/L | ND | 50 | 50 | 47.7 | 47.7 | 95 | 95 | 90-110 | 0 | 20 | |
| Sulfate | mg/L | 829 | 500 | 500 | 1330 | 1330 | 100 | 100 | 90-110 | 0 | 20 | |

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

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QUALIFIERS

Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

PASI-V Pace Analytical Services - Virginia

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

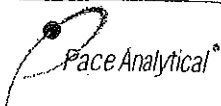
Project: NPDES-LINE 3 Wk1

Pace Project No.: 1260694

| Lab ID | Sample ID | QC Batch Method | QC Batch | Analytical Method | Analytical Batch |
|------------|--------------------------|-----------------|------------|-------------------|------------------|
| 1260694002 | WS-002 Scrubber Make-Up | EPA 200.7 | MPRP/6472 | EPA 200.7 | ICP/4933 |
| 1260694003 | WS-003 Thckener Overflow | EPA 200.7 | MPRP/6472 | EPA 200.7 | ICP/4933 |
| 1260694001 | WS-003 Thckener Overflow | EPA 300.0 | WETA/15597 | | |
| 1260694002 | WS-002 Scrubber Make-Up | EPA 300.0 | WETA/15597 | | |
| 1260694003 | WS-003 Thckener Overflow | EPA 300.0 | WETA/15597 | | |

REPORT OF LABORATORY ANALYSIS

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| | | |
|--|------------------------------------|--|
|  | Document Name: | Document Revised: 23Feb2015 |
| | Sample Condition Upon Receipt Form | Page 1 of 1 |
| | Document No.: F-VM-C-001-Rev.09 | Issuing Authority: Pace Virginia, Minnesota Quality Office |


Sample Condition Upon Receipt

Client Name: _____ Project #: _____

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☒ Client
☐ Commercial ☐ Pace ☐ Other: _____

Tracking Number: _____

WO# : 1260694



1260694

Custody Seal on Cooler/Box Present? ☐ Yes ☒ No Seals Intact? ☐ Yes ☒ No

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☒ None ☐ Other: _____ Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ 140792808 Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 2.7 Cooler Temp Corrected °C: 0.3 Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA

Temp should be above freezing to 6°C Correction Factor: 0.3 Date and Initials of Person Examining Contents: 2-5-16 CL

| Chain of Custody Present? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 1. |
|---|--|--|
| Chain of Custody Filled Out? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 2. |
| Chain of Custody Relinquished? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 3. |
| Sampler Name and Signature on COC? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 4. |
| Samples Arrived within Hold Time? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 5. |
| Short Hold Time Analysis (<72 hr)? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A | 6. |
| Rush Turn Around Time Requested? | <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A | 7. |
| Sufficient Volume? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 8. |
| Correct Containers Used? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 9. |
| -Pace Containers Used? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | |
| Containers Intact? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 10. |
| Filtered Volume Received for Dissolved Tests? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A | 11. Note if sediment is visible in the dissolved containers. |
| Sample Labels Match COC? | <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A | 12. |
| -Includes Date/Time/ID/Analysis Matrix: <u>UT</u> | | |
| All containers needing acid/base preservation will be checked and documented in the pH logbook: | <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A | See pH log for results and additional preservation documentation |
| Headspace in Methyl Mercury Container | <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A | 13. |
| Headspace in VOA Vials (>6mm)? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A | 14. |
| Trip Blank Present? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A | 15. |
| Trip Blank Custody Seals Present? | <input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A | |
| Pace Trip Blank Lot # (if purchased): | | |

CLIENT NOTIFICATION/RESOLUTION

Person Contacted: _____ Date/Time: _____ Field Data Required? ☐ Yes ☐ No

Comments/Resolution: _____

FECAL WAIVER ON FILE Y N TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: XAH for HRZ Date: 2-5-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)